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PRODUCTION OF RUTIN FROM BUCKWHEAT LEAF MEAL

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SUMMARY

Based upon pilot plant experience, a method for the large-scale preparation of buckwheat leaf meal is given. Such meal can be stored for at least 8 months without deterioration and can be used as a source of the new drug rutin. The necessary equipment and details of operation are given for two methods for preparing pure rutin from buckwheat leaf meal. The relative merits of the two processes are discussed, and the factors to be considered in choosing between them are brought out.

Acknowledgment

The authors wish especially to acknowledge the valuable assistance of J. F. Couch, who initiated the studies on rutin in this Laboratory and developed the first practicable extraction process for preparing this substance. Acknowledgment is also due M. J. Copley, C. F. Krewson, J. Naghski, and W. L. Porter, for many valuable suggestions during the course of this work.

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INTRODUCTION

As a result of J. F. Couch's suggestion in 1941 that rutin might reduce capillary fragility, clinical tests were made by J. Q. Griffith, Jr.³ These tests established the value of rutin for this purpose.

Couch originally obtained rutin from high-grade tobacco,⁴ but the high cost of this raw material impelled him to search for other sources. Buckwheat was found to be an economical source. He obtained pure rutin by a process involving the extraction of fresh buckwheat with alcohol.⁵

This circular describes the production of rutin from dried buckwheat leaf meal. Use of the dried buckwheat permits year-round operation, which is not possible when the fresh plant is employed. Furthermore, the amount of alcohol required per pound of rutin is very much less for the dried material. During 1944 and 1945 pilot-plant production of rutin from buckwheat leaf meal was carried out at the Eastern Regional Research Laboratory in the Chemical Engineering and Development Division. Approximately 40 pounds of rutin was produced, and information was obtained as to the type and size of equipment for commercial production.

Two methods were used in producing this rutin. Both are presented in this circular, as the choice between them may well depend on conditions and equipment in a plant already available.

PREPARATION AND STORAGE OF LEAF MEAL

Buckwheat Harvesting

Studies by J. F. Couch⁶ showed that the rutin content of the entire buckwheat plant (calculated on a moisture-free basis) reaches a maximum at a very early stage of growth, when only a few blossoms are yet formed. As the growth of the plant proceeds thereafter, the percentage of rutin decreases, owing in part to the relatively faster growth of the stem tissues, which contain a much smaller percentage of rutin. Therefore, we may assume that the percentage of rutin in the leaf-and-flower portion of the plant, and thus in the dried leaf meal, remains approximately constant

1. HEAD, CHEMICAL ENGINEERING AND DEVELOPMENT DIVISION.
2. CHEMICAL ENGINEER, CHEMICAL ENGINEERING AND DEVELOPMENT DIVISION.
3. GRIFFITH, J. Q., JR., COUCH, J. F., AND LINDAUER, M. A. EFFECT OF RUTIN ON INCREASED CAPILLARY FRAGILITY IN MAN. PROC. SOC. EXPTL. BIOL. AND MED., VOL. 55, NO. 3, PP. 228-9, 1944.
4. COUCH, J. F., AND KREWSON, C. F. RUTIN. UNITED STATES DEPARTMENT OF AGRICULTURE MIMEOGRAPH CIRCULAR AIC-52. (EASTERN REGIONAL RESEARCH LABORATORY) JULY 1944.
5. COUCH, J. F., KREWSON, C. F., AND NAGHSKI, J. PREPARATION OF RUTIN FROM BUCKWHEAT. PRESENTED AT THE MEETING OF THE AMERICAN CHEMICAL SOCIETY IN PHILADELPHIA ON JUNE 13, 1945.
6. COUCH, J. F., NAGHSKI, J., AND KREWSON, C. F. BUCKWHEAT AS A SOURCE OF RUTIN. SCIENCE (IN PRESS).

until the blossoms begin to change into seeds or the leaves begin to atrophy. Couch found that 26 days after planting, when only one to three blossom heads were in bloom, the plant contained 2.98 percent of rutin (on a moisture-free basis), totaling 6.9 milligrams per plant. Seven days later the rutin content was 2.47 percent, totaling 19.1 milligrams, and after another 7 days it was 1.76 percent, totaling 24.2 milligrams. Shortly thereafter the seeds started to set, with a considerable decrease in rutin percentage, though not in total rutin per plant.

From these data it would appear that the buckwheat should be harvested sometime during the blooming period. Since the rutin content of the plant changes so greatly during growth, each harvest should be checked by assays for rutin content. Similarly, if leaf meal is purchased, it should be assayed, because careless drying can seriously diminish the rutin content, as will be shown later. Progressive plantings should be made, so that drying the harvested crop can proceed continuously. In the latitude of Philadelphia it is usually possible for a field to produce three crops per year.

It is very important to dry the harvested plant as quickly as possible, avoiding exposure to the sun or any delay that might cause wilting. Serious loss of rutin, possibly caused by enzyme action, occurs in a few hours if the cut plant is left lying in the field.

Preparation of Buckwheat Leaf Meal

It is assumed that drying the buckwheat plant and preparing leaf meal from it will be done at or near the place of growth and that the meal will be shipped to the rutin manufacturer.

During storage in the dark for 8 months (which is as long as our tests have been in progress) dried buckwheat leaf meal suffered no loss of rutin. These tests included meals of moisture contents between 5.8 and 9.3 percent. It can be safely assumed that a manufacturer can produce rutin steadily throughout the year from meal prepared during the summer.

Drying the leaves and flowers must be rapid to avoid the serious loss of rutin that occurs during slow drying. Couch and Krewson⁵ found that most of the rutin in tobacco is lost in the air-curing process; the behavior of the buckwheat plant is similar. Laboratory tests of Couch, Naghski and Krewson⁶ showed the large loss of rutin that occurs when the plant is completely dried in a laboratory oven at temperatures ranging from 160° to 230°F. However, since the stems of the plant contain little rutin, they can be discarded without drying. By stopping the drying operation as soon as the leaves become dry and by drying under conditions specifically designed to minimize loss, the overall loss of rutin can be reduced to about 20 percent. Discarding 35 percent of the plant as stems gives a meal 23 percent richer in rutin than the plant. The recommended procedure therefore is to dry as quickly as possible, and carry the drying only far enough to embrittle the leaves and flowers; the stems being much thicker, remain moist and tough. The plants in this condition are subjected to mechanical crushing, which breaks the brittle leaves and flowers away from the stems and crushes them into fragments. The fine material is then separated from

the stems by screening. The material in various stages of this preparation is shown in Figure 1.

A flow sheet of the preparation of leaf meal is given in Figure 2. Figure 3 shows the equipment used in our pilot plant. The drier is shown at A, with the chopped buckwheat being fed in at the right. The dried plant emerges at the lower left, B, and is carried by the conveyors to the trommel, C. The stems pass out from the end of the trommel to the chute, F, and are discarded. The fragmented leaf material, passing through the mesh of the trommel, falls at D to the vibrating screen. The three fractions from the screen are collected in containers in the cabinet E.

DRYING - The most suitable type of drier for drying the plant in this manner is one in which moderately heated air is forced at high velocity through a shallow bed of the material supported on a perforated belt or belts moving continuously through the heating chamber. The rotary kiln type of drier sometimes used for alfalfa in which the current of air carries the material through the drying zone would probably be unsuitable for the preparation of leaf meal by the separation of fractionally dried material, as described above. This type of drier has a tendency to produce material uniformly dried throughout. Such driers, however, generally employ high temperatures in the entering gases and might be satisfactory for the very rapid drying of buckwheat without undue destruction of rutin. The product could then be ground to a whole meal somewhat less rich in rutin than leaf meal but possibly satisfactory as a source of rutin. The possibility of adapting rotary and other conventional types of alfalfa driers to the production of buckwheat meals and leaf meals is under investigation.

The drying time can be considerably decreased and the desirable uniformity of product insured by turning the plant over after it has been partly dried. This breaks up compacted mats of wilted material within the bed, and moves the less-dried lower material to the more favorable top position, where it receives hotter air. In a multiple-belt drier, this is done by dropping the buckwheat from one belt to another running beneath it. A three-belt drier is recommended; this turns the material over twice. The belts should be constructed of flexible wire mesh (approximately 7 to 10 mesh). The air should be uniformly distributed down through the material at a rate of at least 175 cubic feet per minute per square foot of belt. Suitable heating units with controls should be provided, to maintain the air at 190°F. The drier should also be provided with an automatic variable-speed feeding device and rotating "doffers" having fingers to break up the mat of material as it drops from one belt to another.

Normally, the second belt is run at one-half the speed of the first, and the third at one-half that of the second, but the speed of each belt relative to the others should be adjustable. The range of adjustment of the speed of the first belt especially should be liberal, as for best drying results buckwheats of different moisture contents and different maturities require different treatments on the first belt. For plants in full bloom and containing 81 percent moisture, the initial loading on the first belt should be not more than 2 pounds (fresh weight) per square foot; if too deep a layer is used the buckwheat when wilted by partial drying settles down to a compact mat that obstructs the passage of air. Under normal operating conditions, the air pressure drop through the bed is about 0.4 inch

of water. Younger plants, or those having a much higher moisture content (for example, 86 percent) or surface moisture, require lighter loading on the first belt, as they are more prone to wilt and form compact mats. This change in loading is accomplished by increasing the speed of the first belt relative to the others.

For the purposes of this circular, we have made the following assumptions as to size of the factory, characteristics of the buckwheat, and operating data:

Yearly output of rutin, pounds	10,000
Days (24 hours) of operation of rutin factory	250
Days (24 hours) of operation of drier	50
Moisture content of buckwheat, percent	86
Rutin content of buckwheat, moisture-free basis, percent	2.44
Loss of dry matter as stems, percent	35
Moisture content of leaf meal, percent	8.00
Loss of rutin in drying and separating stems, percent	20
Recovery of the rutin in the leaf meal (by either hot-water process or dilute alcohol process), percent	70

The following data have been calculated from these figures:

Rutin content of leaf meal, percent moisture-free basis	3.00
Daily output of rutin, pounds	40
Daily consumption of leaf meal by rutin factory, (8 percent moisture) pounds	2,070
Daily output of leaf meal (8 percent moisture) by drier, pounds	10,350
Daily consumption of fresh plant by drier, pounds	104,500

To dry during the harvest (10 weeks, 5 days a week) enough buckwheat to supply the factory with meal for a year, according to these assumptions the drier would have to handle 104,800 pounds of fresh plant of 86 percent moisture content per day of 24 hours, or 77,200 pounds if the moisture content were 81 percent. Young plants, or in a rainy season plants in full bloom, may easily contain 86 percent moisture; therefore we have used this figure in calculating the required size of drier. For the reasons already stated, it is not feasible to attempt to economize on drying by letting the plant dry even slightly in the field. The belts should have a total drying area of 700 square feet. Three belts 8-1/2 feet wide, each having an active length of 27 feet, would be suitable. This drier could be arranged with each belt traveling back underneath the preceding one or with all three traveling ahead in the same line. With air at 190°F., the total drying time will be about 30 minutes but will vary according to the characteristics of the buckwheat. In a drier of these dimensions, this figure corresponds to a speed of about 1.6 feet per minute for the third belt and 3.2 for the second. The first belt should have a range of adjustment of speed from 4 to 10 feet a minute or thereabouts; if run at 6.4 feet per minute, the loading to handle 104,800 pounds per day will be 1.34 pounds per square foot. The drier would probably require about 80 horsepower for fans and drives; if steam heated, it would consume approximately 7500 pounds of steam per hour.

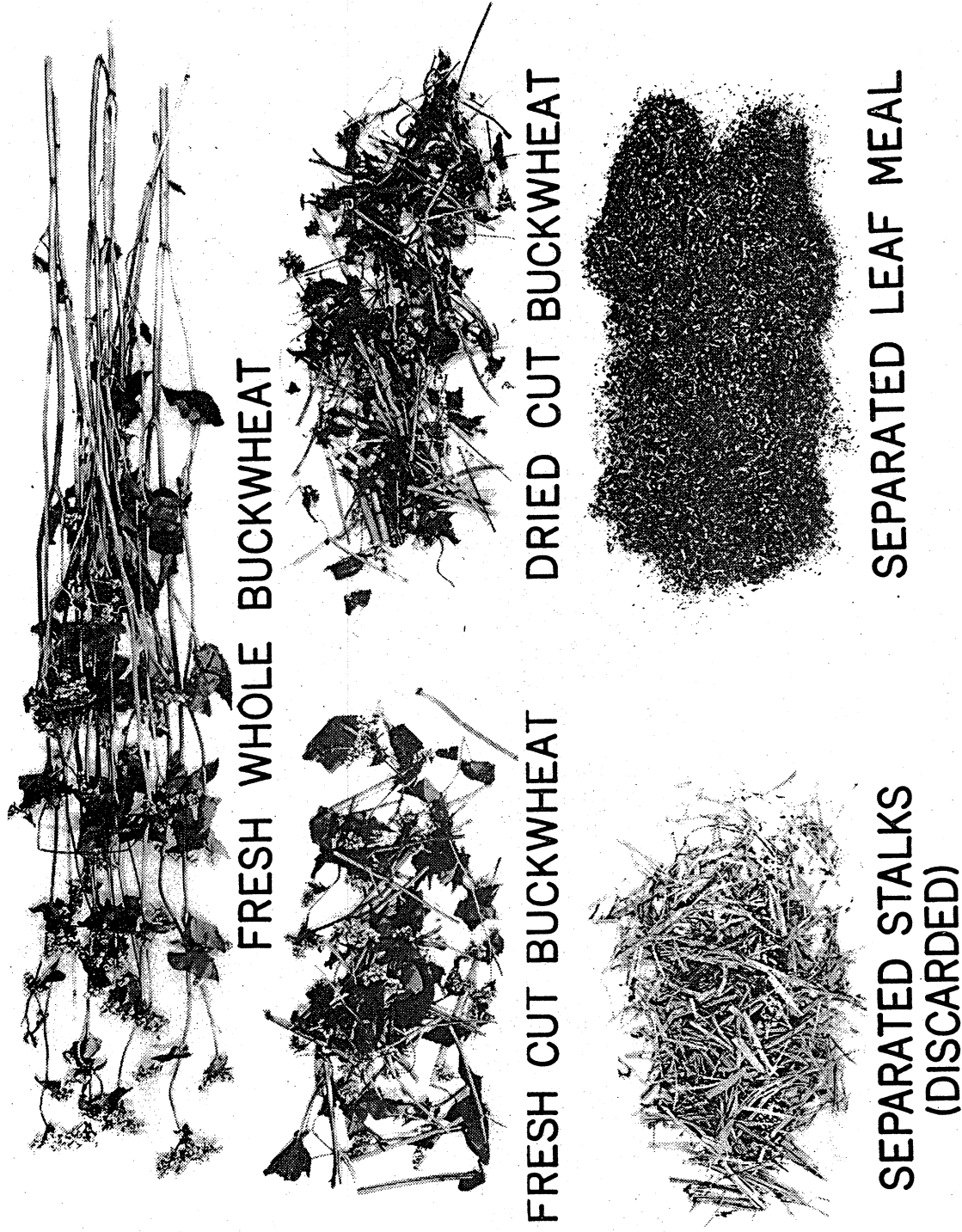
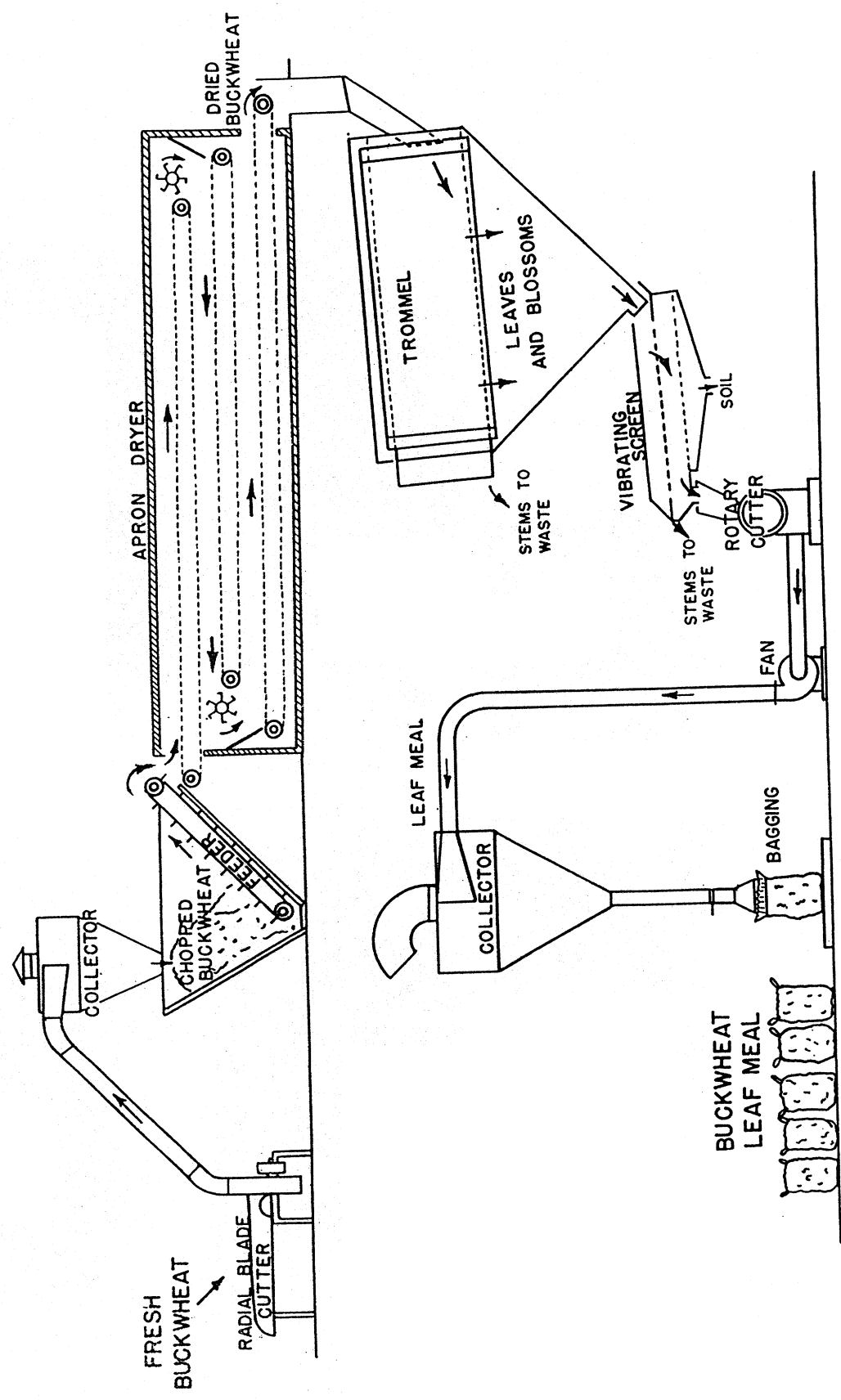


FIG.-1- VARIOUS STAGES IN THE PREPARATION OF
BUCKWHEAT LEAF MEAL

FIG. 2
 FLOW DIAGRAM FOR PREPARATION OF
 BUCKWHEAT LEAF MEAL



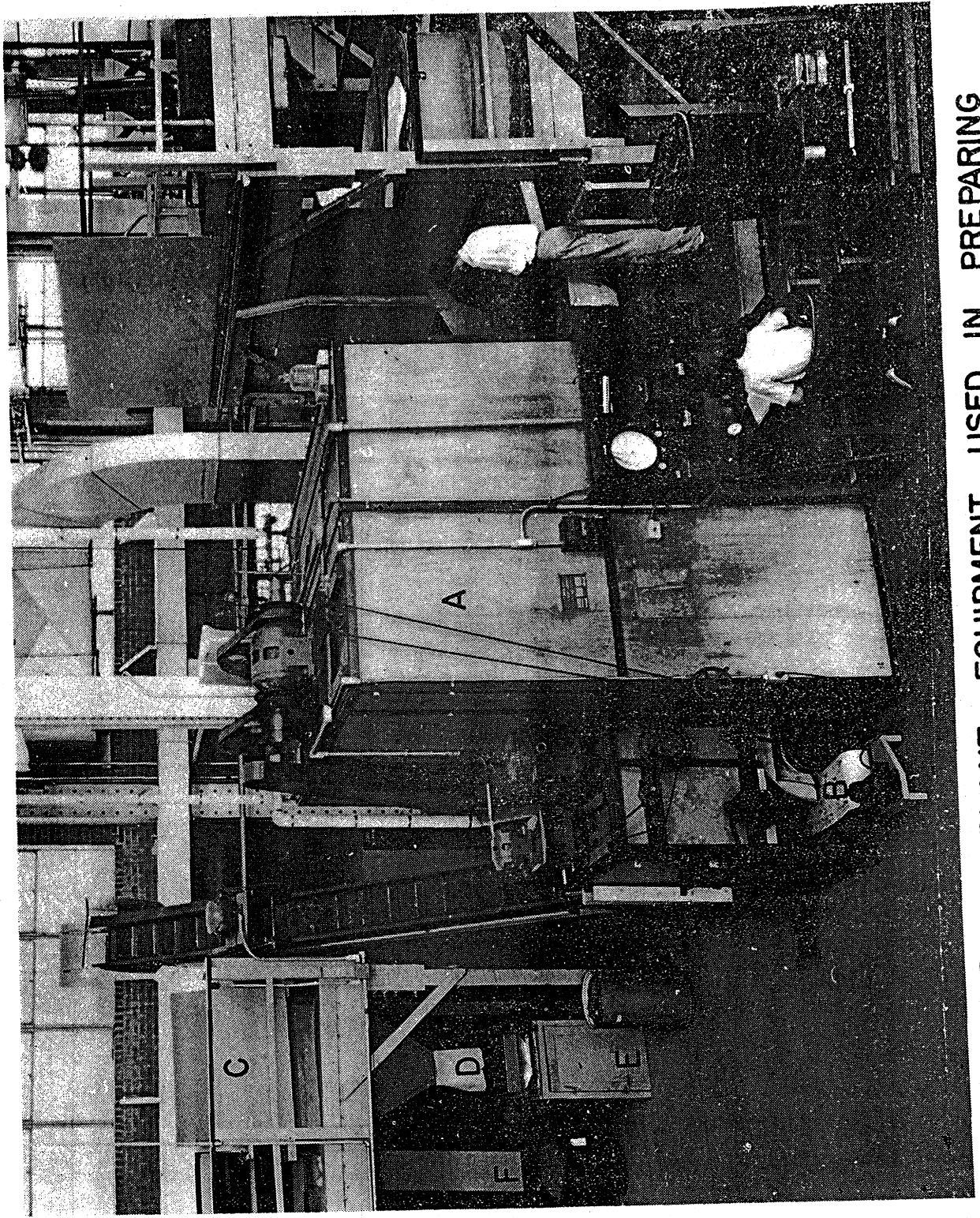


FIG.-3- PILOT PLANT EQUIPMENT USED IN PREPARING
BUCKWHEAT LEAF MEAL

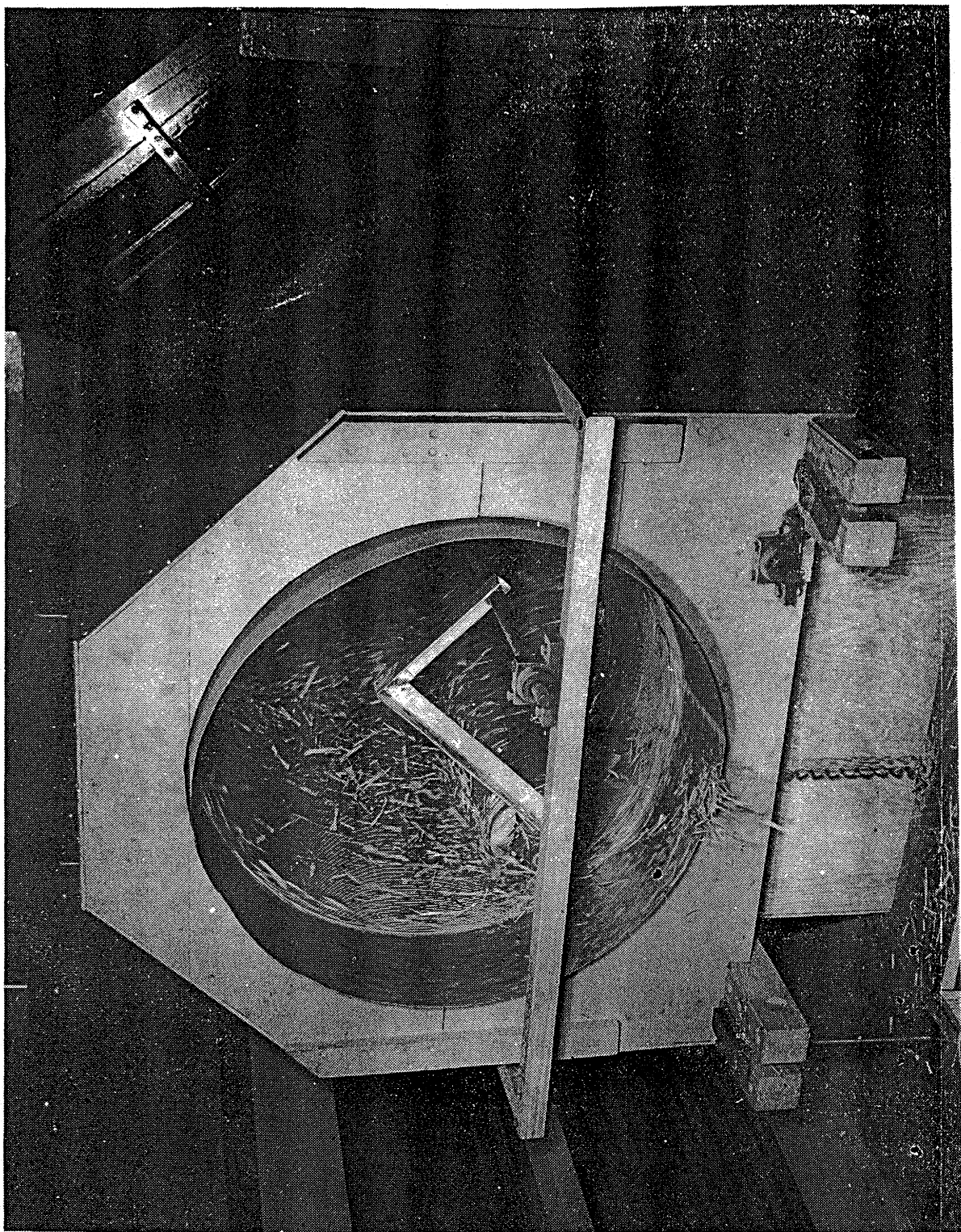


FIG.-4-DISCHARGE END OF TROMMEL USED IN
SEPARATING BUCKWHEAT STEMS FROM LEAVES

To enable the buckwheat plant to be loaded easily and uniformly on the drier belts, it should be chopped coarsely and with a minimum of crushing or bruising. An ensilage cutter of the radial blade type, set to produce pieces about 2 inches long, is suitable. If the chopped plant is sent to the drier within a few minutes, no appreciable loss of rutin occurs, but no delay nor storage of the chopped material can be tolerated.

SEPARATION AND GRINDING - To separate the leaf and flower material, now dry and brittle, from the still moist stems, the output of the drier is fed into a rotating screen, or trommel, provided with devices for breaking up the brittle material. This operation must be carried out immediately; otherwise the moist stems will rehydrate the leaves and separation will be impossible. A continuous trommel 45 inches in diameter by 14 feet long handles the output of the drier adequately. It should be made of heavy wire mesh having $3/16$ inch openings, attached to the inside of a cage (which can be of wooden strips) to form a smooth inside surface without projections. The speed should be 36 revolutions per minute for a screen of this diameter; if another size is substituted, the speed should be in proportion to the square root of the diameter. This speed carries the material up nearly to the top of the drum and throws it down across the drum. The end-to-end slope of the drum should be adjustable to give a proper flow of material from the feed end to the discharge end. Normally, the material should travel through the drum in 2 or 3 minutes.

Any mechanism that breaks the brittle portions into small fragments, either before entering the trommel or while inside it, can be used. One scheme found satisfactory comprises a line of heavy wooden rollers, about 6 inches in diameter and 2 or 3 feet long, mounted in the trommel, riding on the inside surface of the screen-drum. Each end of the axle of each roller rotates in a bearing on the end of an arm. To explain the relative location of the parts, let us look at the discharge end of the trommel and imagine it to be a clock dial (see Figure 4). The drum rotates clockwise. The point of contact between the rollers and the drum is at 8 o'clock. The arms are all pivoted on a fixed shaft located near the inner surface of the drum, at 3 o'clock. Thus the relative motion of the parts is such that the weight of the roller wedges it against the screen, producing a combined crushing and rubbing action on the plant material as it is carried between the screen and the roller. The fragmented material then passes through the mesh of the trommel drum, and the stems pass out through the open end. Figure 4 is a photograph of the discharge end of a trommel arranged as described above.

The fragmented material from the trommel is not yet clean. It contains stem pieces that passed endwise through the mesh of the trommel, and of course all the earth that might have been on the harvested plant. It is therefore sent to a two-deck (self-cleaning) vibrating screen having a 3-mesh and a 60-mesh sieve. The former holds up most of the stem pieces, and the latter permits the dirt to pass away. A screen area of about 20 square feet in each deck should be adequate.

The leaves then pass directly, for final chopping to form a leaf meal of proper size for extraction, through a magnetic separator to a high-speed rotary cutter. It is not necessary to do this chopping immediately, but

obviously it is simpler to do so, feeding the output of the screen directly to the chopper. A cutter having three or four straight knives arranged to form a cylinder rotating inside a cage provided with four or five stationary straight knives has been found satisfactory. The knives run at extremely close clearance. The lower half of the cage is composed of a perforated metal screen; for this work 1/4-inch round openings are satisfactory. A machine having a knife cylinder about 9 inches in diameter by 20 inches long should be adequate; for this light service a 3 horsepower motor should suffice.

A hammer-mill is not recommended for this work, because its product would contain too great a proportion of fines, which clog the extraction tanks and prevent good drainage of the liquid from them.

Suggested List of Equipment

Binder-reaper and tractor	\$ 1,700
Truck	1,500
Ensilage cutter	300
Drier, 700 square feet	30,000
Trommel	900
Two-deck screen	300
Meal cutter	1,500

HOT-WATER EXTRACTION PROCESS

General Description

In the hot-water extraction process, buckwheat leaf meal prepared as described in the foregoing section is given three 20-minute extractions in boiling water to remove the rutin. This recovers approximately 97 percent of the rutin. After the extracts are filtered, they are concentrated by vacuum evaporation. Proteins and colloidal materials are coagulated by adding an equal volume of 95 percent ethyl alcohol⁷ to the concentrates. The curd so formed is removed by filtration; the filtrate is evaporated with addition of water until practically all the alcohol is driven off. This water solution is permitted to crystallize, and the crude rutin is filtered off. A recrystallization is then given. If traces of alcohol-insolubles are objectionable in the finished rutin, they may be removed at this point by dissolving the rutin in a small amount of hot alcohol and filtering; thereafter in a vacuum evaporation step the alcohol is replaced by water. A third and final crystallization is then given to insure the desired purity, after which the crystals are filtered off and dried. Figure 5 is a flow diagram of the process.

One of the obvious advantages of this process is that no alcohol is used in the extraction and only a small quantity in the purification, and hence no large rectification equipment is necessary. Extraction with hot water is also very rapid, the total extraction cycle being only about 3 hours, as contrasted with much longer extraction periods required with alcohol. A further advantage is that fats and tarry materials are left in the marc;

7. WHEREVER ALCOHOL IS MENTIONED IN THIS CIRCULAR, BENZOL-DENATURED ALCOHOL IS MEANT. ALL CONCENTRATIONS ARE EXPRESSED BY VOLUME.

hence benzol with its attendant hazards is not required in rutin purification.

Details of Operation

The operations described here are those required in a plant producing 40 pounds per day of pure dry rutin from buckwheat leaf meal containing 3 percent rutin on the moisture-free basis. This is equivalent to 10,000 pounds in an operating year of 250 days. The equipment described is that needed in a new factory designed for the express purpose of producing rutin. It is recognized, however, that in most cases rutin will be produced in equipment already available in pharmaceutical plants. Much of such equipment will undoubtedly be adaptable to the rutin process.

Dissolved iron, even in very small quantity, imparts a brown discoloration to the finished rutin. Hence, to avoid the special procedures required to remove the iron, equipment that might cause contamination should not be used. The only metal used in our pilot-plant was 18-8 stainless steel, which was satisfactory. No definite data on other metals are yet available.

EXTRACTION- The extraction is carried out in one wooden tank of approximately 600 gallons capacity, equipped with a false bottom covered with a 30-mesh stainless steel screen. Into this are charged 259 pounds of leaf meal (8 percent moisture) and 485 gallons of water; this is equivalent to 238 pounds moisture-free weight of meal. Slow speed agitation should be supplied to insure intimate mixing, and a small amount of steam should be introduced to offset radiation losses. The main supply of hot water for extraction should be derived from a separate hot water heating system capable of heating approximately 485 gallons of water to boiling every 30 minutes.

Extraction of rutin from dried leaf meal with hot water is so rapid that equilibrium is attained in 20 minutes or less.

Since equilibrium conditions (balanced distribution of rutin between leaf meal and liquor) are obtained in 20 minutes or less, there is no advantage in increasing the leaching time. Our pilot-plant experience showed that three extraction treatments of 2 hours each did not significantly alter the overall yield of rutin. Some reduction in yield might have been anticipated because of rutin's reputed sensitivity to heating in water solution. Prolonged extraction of leaf meal with boiling water is inadvisable, being both unnecessary and possibly conducive to subsequent filtration troubles.

By using in each extraction a ratio of 17 pounds of water per pound (moisture-free weight) of leaf meal and giving three 20-minute extractions, a complete extraction cycle can be accomplished in 3 hours. This will remove approximately 97 percent of the rutin if thorough drainage is obtained. Good draw-off will result if the extracts are withdrawn through the false bottom with a positive suction rotary pump. Pilot-plant experience has shown that the extract removed is approximately 74 percent of the total liquid present. Therefore for the second and third extractions of each cycle, 74 percent of 485 gallons, that is, 358 gallons of water is added.

The exhausted marc is easily removed from the extraction tank if a connection is provided from the false bottom to the drain, into which the marc can be flushed with a hose.

CONCENTRATION OF EXTRACTS - Before concentration, and the subsequent coagulation step, which employs alcohol, the extracts are filtered to remove fine entrained leaf meal and thereby avoid having any fats extracted by the alcohol from such entrainment. A cypress plate and frame filter press having 75 square feet of filtering surface can be used for this purpose. A fine twill is satisfactory for dressing. For flexibility of operation, it is desirable to have a wooden storage tank of approximately 500 gallons capacity between the leaching tank and the filter press. Approximately 7 pounds of filter aid should be added to the tank each time a draw-off from the leaching tank is added. The tank should be agitated and equipped with stainless steel heating coils to keep the solution hot and prevent any crystallization of rutin before filtration. Ordinarily three leaches, constituting one cycle, can be filtered without redressing the press.

The extracts are concentrated in a stainless steel vacuum evaporator with approximately 75 square feet of heating surface. Since the formation of a good filterable curd in the next step is contingent upon evaporating to the proper degree, the evaporation end point must be controlled. Viscosity is a convenient criterion. Evaporation can desirably be carried to the maximum degree at which the slurry is still fluid enough to flow from the evaporator, since the further evaporation is carried, the less alcohol is required in the subsequent curd-forming step. This corresponds approximately to a reading of 1000 centipoises when measured at 106° F. with a Brookfield viscosimeter using a No. 3 bob at 30 revolutions per minute.

In order to avoid the possible destruction of rutin through prolonged heating, it is desirable to operate the evaporator batchwise, concentrating the extracts from one extraction cycle (3 hours) at a time. For convenience, the concentrates can be accumulated in a wooden tank of approximately 400 gallons capacity.

COAGULATION AND REMOVAL OF NONRUTIN SOLIDS - The concentrates accumulated over a 24-hour period are put into a coagulation tank, where an equal volume of 95 percent ethyl alcohol is added and the mixture is brought to the boiling point to insure complete solution of the rutin. One minute of boiling is usually sufficient for this. The tank may be of wood and should have a capacity of about 800 gallons. It must be equipped with an agitator and stainless steel coils for heating and cooling. In order to prevent loss of alcohol during heating, the tank should be tightly covered and equipped with a small reflux condenser. Although our experience has shown that an easily filterable curd is obtained when concentration and coagulation are carried out as described above, in the interests of good control a small sample of the slurry should be removed and filtered through paper into a test tube. If filtration is not rapid and the filtrate not sparkling clear, a small amount of additional alcohol will insure the desired result.

The curd formed in the coagulation step is removed by filtering in a cypress filter press having about 50 square feet of filtering area. A fine twill cloth may be used for dressing the press. Experience has shown that the most rapid filtering is obtained if the pressure is not permitted to rise above approximately 20 pounds per square inch.

In order to obtain the maximum yield of rutin, it is obviously desirable to wash the curd while still in the filter press. For this purpose, alcohol of about 50 percent strength should be used as it has a greater solvent action for rutin than 95 percent alcohol.

After removal of the curd, the filtrate is sent to a stainless steel evaporator having approximately 10 square feet of evaporating surface. In this step, the alcohol is driven off and replaced with water. The alcohol so recovered must be rectified for reuse. The alcohol used in this step is equivalent to approximately 7.8 gallons of 95 percent alcohol per pound of rutin. The evaporation should be carried out stepwise to minimize the time in which the alcoholic rutin solution is kept hot. To make this possible a holding tank of about 400 gallons capacity should be located between the filter press and the evaporator.

CRYSTALLIZATION OF CRUDE RUTIN - The water suspension of rutin crystals from the small evaporator is pumped to one of three crystallizing tanks. These should have a capacity of about 1400 gallons and should be equipped with stainless steel coils for heating and cooling. Agitation should be provided to accelerate solution and heat transfer. Enough hot water is added at this point to dissolve the rutin. In pure hot water the solubility of rutin is 0.55 percent; actually at this stage the presence of sugars increases the solubility, so that less water is required and hence less rutin lost in the mother liquor. Cold water is then drawn into the coils and the tank cooled down in a short time, for example, 2 hours. The contents of the tank are allowed to stand for approximately 44 hours without agitation, at the end of which time the crude rutin crystallization is complete. A shorter crystallization time than this at this stage may result in loss of uncrystallized rutin. As a matter of precaution, a plain tank should be provided for holding the mother liquor for an extra day to see whether any further crystallization occurs.

The crude crystals are recovered by filtering the slurry through a stainless steel filter press having approximately 100 square feet of filtering surface. This can be conveniently accomplished in five cycles, totaling about 8 hours, thereby leaving this press free for use in obtaining crystals from two successive purification stages. A fine twill cloth is satisfactory for dressing the press.

The crude crystals are redissolved in hot water in a tank equipped with an agitator and stainless steel coils and having a capacity of about 1200 gallons. Since rutin is converted to quercetin in alkaline solution, this water must be adjusted to a pH below 7.0. This may be done simply by adding a small amount of sulfuric acid. At this point approximately 1 pound of silica gel is added per pound of rutin in the tank, in this case approximately 50 pounds. The purpose of this is to adsorb chlorophyll and certain other constituents undesirable in finished rutin. There should also be added about 5 pounds of some filter aid, such as diatomaceous earth. After thorough agitation the hot solution is filtered through a stainless steel steam-jacketed filter press having about 40 square feet of filtering surface. In this filtration, the twill cloths are preferably covered with paper for greater clarification. The hot filtrate should be discharged into a wooden crystallizing tank of about 1200 gallons capacity, similar to the one used in the

preceding crystallization. In this case approximately 22 hours are required for complete crystallization. The crystals from this operation are then filtered off; the same 100-square-foot filter press employed in obtaining the first crystals is used.

REMOVAL OF ALCOHOL INSOLUBLES - If traces of alcohol-insoluble materials are to be removed from the rutin, it is customarily done at this stage. The crystals are removed from the press, dried, and dissolved in 1000 pounds of hot 95 percent alcohol. Drying can be done in the same unit used for drying the finished rutin. An alternate procedure can be used when time or facilities for drying at this stage are not available. This entails blowing air through the filter press before removing the crystals in order to reduce their moisture content to about 60 percent, and then dissolving the moist crystals in 1000 pounds of hot anhydrous alcohol. In either case a 200-gallon stainless steel jacketed kettle equipped with a tight cover and a small reflux condenser is suitable. The rutin does not go completely into solution until the boiling point is reached and maintained for about 5 minutes. This hot solution is then filtered through the same steam-jacketed stainless steel filter press used in filtering the hot aqueous rutin solutions. Filtration is rapid even when papers backed by cloths are used.

The hot alcoholic filtrate is discharged from the press into the same 10-square foot evaporator used for driving off the alcohol after the curd filtration. In the evaporation, most of the alcohol is recovered at 95 percent; the remainder requires rectification. This evaporation operation is conveniently done batchwise under vacuum to minimize the time of holding rutin in hot alcoholic solution.

FINAL CRYSTALLIZATION AND DRYING - The butts from the foregoing evaporation consist of an aqueous suspension of rutin crystals. They are accumulated in a stainless steel, steam-jacketed, agitated, final dissolving tank, where a sufficient quantity of hot water (pH kept below 7.0) is added to dissolve the rutin. The capacity of this tank should be approximately 1000 gallons. There should be a little more than 40 pounds of rutin present at this stage, requiring about 950 gallons of total water (based on a solubility of 0.55 gram of rutin per 100 grams of boiling water). Here again 50 pounds of silica gel is added to the solution. Five to ten pounds of filter aid may also be used to expedite filtration. This solution is filtered hot through the same steam-jacketed stainless steel filter press employed in previous hot aqueous and alcoholic filtrations. Paper backed with twill cloth should be used. The filtrate is discharged to another stainless steel steam-jacketed agitated tank, where it is cooled down promptly, for example, in 2 hours, and permitted to crystallize for at least 12 hours.

The crystals are recovered by filtering the cold solution through the same press previously employed for crude crystal filtrations. With proper scheduling of operations, ample time is available for a thorough cleaning of this press to remove traces of crude rutin before this final crystal filtration. While the crystals are in the press, they should be washed with cold filtered water having a pH slightly below 7.0.

The final rutin crystals from the press may be dried in a tray drier either under vacuum or at a temperature not exceeding 266° F.

YIELD OF RUTIN - The overall yield of pure rutin, based on the rutin content of the leaf meal on a moisture-free basis, has been conservatively assumed to be 70 percent. Actually, pilot-plant and laboratory experiments show that with careful operation overall yields between 75 and 80 percent may be obtained. Factors contributing to good yield are thorough washing of the curd in the filter press, allowing sufficient time for complete crystallization, and avoiding prolonged heating of solutions of rutin in alcohol or water.

The extraction cycle recommended should remove about 97 percent of the rutin from the meal. The principal losses occur in the mother liquors from the three crystallizations. The loss is largest in the first mother liquor, where the solubility of rutin is probably increased by the presence of sugars. Attempts to recover additional rutin by evaporation of the mother liquors have not been entirely successful.

WATER SUPPLY - A supply of hot water for extracting the leaf meal can conveniently be had by equipping a wooden tank about 5 feet in diameter by 7 feet deep with automatic level and temperature controls. Heating can be done with open steam, but precaution must be taken to insure that the steam is free from oil.

The hot water used for the first and second dissolving stages on crude rutin can also be heated with open steam; the same precaution should be used. As explained above, the pH must be reduced slightly below 7.0, as hot alkaline water in contact with rutin may convert it to quercetin.

The hot water used in the final crystal solution step should be heated in a jacketed tank. Its pH should also be reduced below 7.0, and it should be passed through a filter.

Suggested List of Equipment

	COST
WATER-HEATING SYSTEM FOR EXTRACTION - Wooden tank, 5 feet in diameter by 6 feet deep, with open steam line. Approximately 800 gallons capacity. Must heat 425 gallons of water to boiling in 30 minutes. Must discharge this amount of water to leaching tank by gravity in 15 minutes.	\$ 350
LEACHING TANK - Wooden tank, 4-1/2 feet in diameter by 5 feet deep, with false bottom covered with 30-mesh stainless steel screen. Approximately 600 gallons capacity. Open steam in tank to offset radiation losses. Holds 485 gallons of water and 233 pounds (moisture-free basis) of leaf meal, which contains 7.14 pounds of rutin.	350
POSITIVE DELIVERY ROTARY PUMP FOR DISCHARGING LEACHING TANK - Must handle 360 gallons of hot water in 20 minutes; 1 horsepower motor.	225
STORAGE TANK FOR EXTRACTS - Wooden tank, 4 feet in diameter by 5 feet deep. Five hundred gallons capacity. Holds extracts while filter press is being redressed.	250

	COST
FILTER PRESS FOR EXTRACTS - Cypress construction; 75 square feet of filtering area.	\$ 625
VACUUM EVAPORATOR FOR CONCENTRATION OF EXTRACTS - Stainless steel evaporator with jet condenser. Must evaporate 1040 gallons of water each 3-hour cycle, that is, 347 gallons per hour. Evaporative surface is 75 square feet if of effective design.	5,000
HOLDING TANK - Wooden tank, 4 feet in diameter by 4 feet deep. Approximately 400 gallons capacity. Used to hold butts from evaporator to avoid holding concentrates in evaporator for too long a period.	100
COAGULATION TANK - Wooden tank, 5 feet in diameter by 6 feet deep, with stainless steel heating and cooling coil and agitator. Eight hundred gallons capacity. Must heat 616 gallons of 50 percent alcohol to boiling point; must cool to room temperature in 1 hour. Must have a small reflux condenser to prevent loss of alcohol.	800
FILTER PRESS FOR CURD - Cypress construction; 50 square feet of filtering area. Must handle 616 gallons of solution in 24 hours.	500
HOLDING TANK - Wooden tank, 4 feet in diameter by 4 feet deep. Four hundred gallons capacity. Holds filtrate from preceding operation while the succeeding operation is being carried on batchwise.	100
SMALL EVAPORATOR - Stainless steel evaporator; condenser and receiver may be copper. Ten square feet of evaporating surface required. Used to remove in eight 2-hour cycles the alcohol from 616 gallons of the foregoing filtrate, which consists of approximately 50 percent alcohol. Also used in a later operation, where 95 percent alcohol is replaced by water.	1,750
FIRST DISSOLVING AND CRYSTALLIZING TANKS - Three wooden tanks, 6 feet in diameter by 6-1/2 feet deep. Fourteen hundred gallons capacity. Equipped with stainless steel coils having sufficient surface to cool the contents from 212° to 80° F. in 2 hours. Agitated for rapid cooling. 3 at \$900	2,700
CRYSTAL FILTER PRESS - Stainless steel backwashing type filter press with 100 square feet of filtering area. Used for 7-1/2 hours each day for filtering first rutin crystals, 6 hours each day, (3 cycles, 2 hours per cycle), for filtering second rutin crystals, and 5 hours each day for filtering final rutin crystals. This permits an interval of 5-1/2 hours for the thorough cleaning of the press necessary before using it for final crystals.	3,000
HOLDING TANK FOR MOTHER LIQUOR - Wooden tank, fourteen hundred gallons capacity.	300

SECOND DISSOLVING TANK - A tank of the same size and construction as the first dissolving and crystallizing tanks.	COST \$ 900
SECOND CRYSTALLIZING TANKS - Two tanks of the same size and construction as the first dissolving and crystallizing tanks. 2 at \$900	1,800
FILTER PRESS FOR HOT FILTRATIONS - Stainless steel steam-jacketed filter press, with 40 square feet of filtering area. Used for filtering the hot-water solutions of the first and second crystals and also later for filtering the hot alcoholic solutions of rutin and removing alcohol insolubles.	2,500
TANK FOR DISSOLVING RUTIN CRYSTALS IN ALCOHOL - Stainless steel jacketed, agitated kettle, 3 feet in diameter by 4 feet deep, with reflux condenser. Two hundred gallons capacity.	1,500
DISSOLVING TANK FOR CRYSTALS IN THE BUTTS AFTER REMOVAL OF STRONG ALCOHOL - Wooden tank, 5-1/2 feet in diameter by 5-1/2 feet deep. One thousand gallons capacity. Stainless steel coil.	800
TANK FOR THIRD AND FINAL CRYSTALLIZATION - Stainless steel, agitated, jacketed tank, 5-1/2 feet in diameter by 5-1/2 feet deep. One thousand gallons capacity.	3,000
SYSTEM FOR SUPPLYING HOT WATER FOR DISSOLVING CRYSTALS FOR FIRST AND SECOND CRYSTALLIZATIONS - Wooden tank, 6 feet in diameter by 5-1/2 feet deep. Heated with open steam, and equipped with automatic temperature and level control. Eleven hundred gallons capacity.	400
SYSTEM FOR SUPPLYING CLEAN, FILTERED, HOT WATER FOR DISSOLVING CRYSTALS FOR FINAL CRYSTALLIZATION - Wooden tank, 5-1/2 feet in diameter by 5-1/2 feet deep, with stainless steel coils. One thousand gallons capacity. Equipped with delivery pump and stone filter in discharge line. Automatic temperature and level controls.	1,200
RECTIFICATION STILL - For rectification of approximately 540 gallons a day of approximately 60 to 95 percent alcohol.	3,500
TRAY DRIER - For removing water from purified rutin.	600

DILUTE-ALCOHOL EXTRACTION PROCESS

General Description

Buckwheat leaf meal prepared as previously described may also be extracted with dilute alcohol. Pilot-plant experiments have shown that alcohol of approximately 70 percent strength is a good concentration to use and a much better extracting agent than 95 percent. A method for manufacturing rutin in which dilute alcohol is used as the extractant is described here because some of the subsequent purification steps are simpler than those required for hot water extraction, and it may be found in some cases that available facilities are more applicable to the dilute-alcohol process. In the extraction with dilute alcohol, the meal is given three 12-hour extractions at room temperature, a ratio of 6 parts of alcohol to 1 of leaf meal (moisture free basis) being used. If circulation of the extract were employed, 6 to 8 hours would be sufficient time for each extraction. The extracts are then concentrated by vacuum evaporation. Since fats and tars are extracted from the meal by dilute alcohol, they must be removed. The bulk of these can be eliminated by hot filtration. The filtrates are then purified by the same process used in hot-water extraction with three exceptions.

- (1) The steps necessary for curd removal are omitted.
- (2) An additional drying step on the purified rutin is required.
- (3) The dried pure rutin must be extracted with benzol to remove the last traces of fats, which are not completely eliminated by filtration.

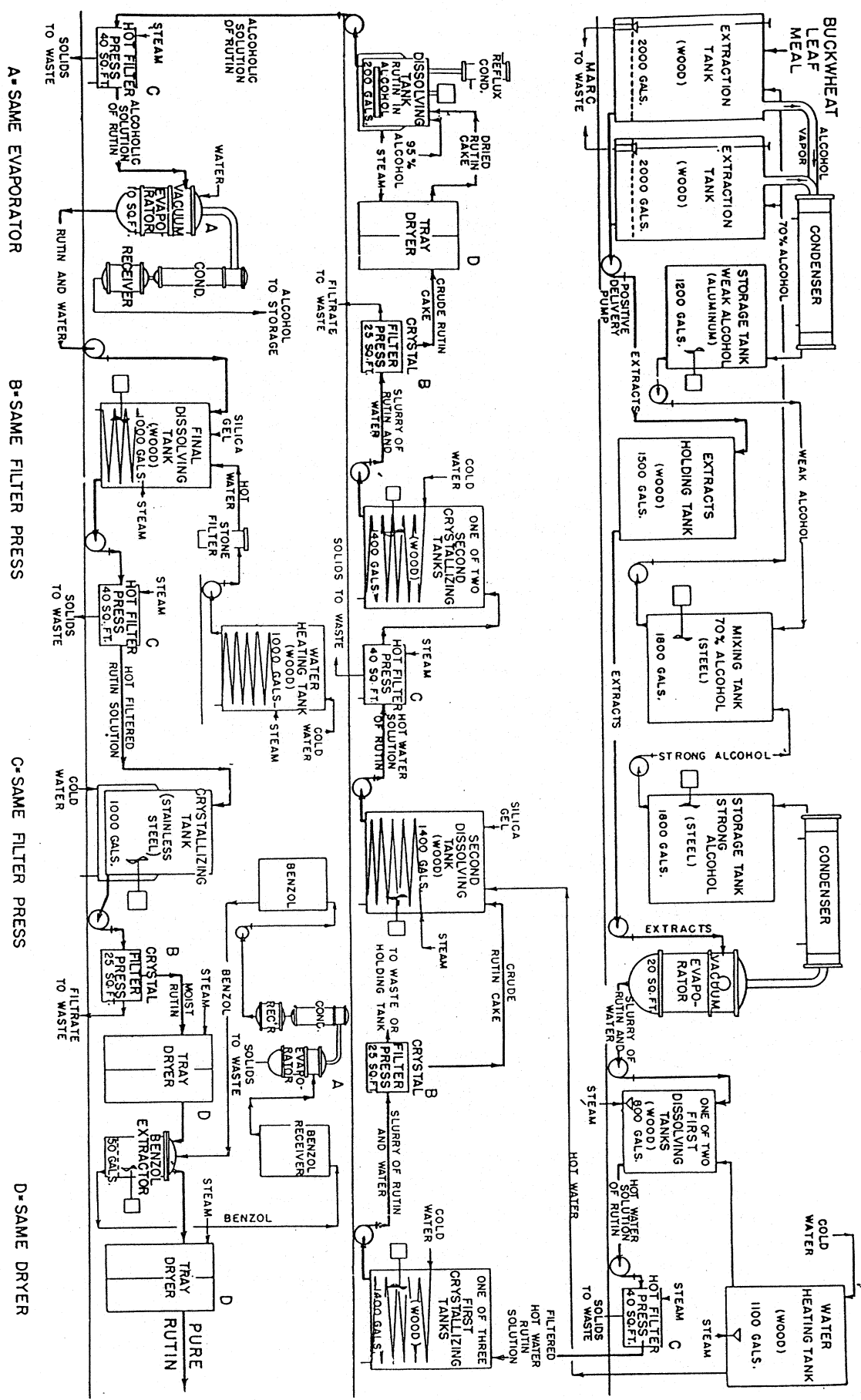
Figure 6 is a flow diagram of the process.

Details of Operation

As in the hot-water process, the basis in this case is a daily production of 40 pounds of pure dry rutin from buckwheat leaf meal containing 3 percent rutin on the moisture-free basis. This is equivalent to 10,000 pounds of rutin per operating year of 250 days. The equipment described is that needed in a new factory designed for the express purpose of producing rutin. In many cases rutin will be produced in pharmaceutical plants already established and equipped with facilities, some of which can be adapted to the dilute-alcohol extraction process. As in the hot-water extraction process, precautions must be taken to avoid iron contamination.

EXTRACTION - Two wooden tanks, each having a capacity of 2000 gallons, are required for the extraction. Each tank should be equipped with a false bottom covered with 30-mesh stainless steel screen, below which is a steam sparger for the introduction of open steam to be used in stripping the alcohol from the marc at the end of each extraction cycle. No agitation is required in the tanks. They should be tightly covered, and both should be connected with a vapor line to a condenser and receiver for the recovery of dilute alcohol from the marc. Both tanks should also be connected to a single positive delivery rotary pump to insure the maximum drainage after each leach.

FIG. 6
 FLOW DIAGRAM FOR PRODUCTION OF RUTIN FROM BUCKWHEAT LEAF MEAL
 BY DILUTE ALCOHOL EXTRACTION PROCESS



With a ratio of six parts of alcohol to one of leaf meal, three 12-hour extractions remove about 95 percent of the rutin. If a ratio of seven of alcohol to one of leaf meal were used, this would be increased to slightly more than 97 percent removal, but it is questionable whether the improved recovery would justify the use of the extra alcohol. The operation of the two extraction tanks is staggered. Each would be charged with 2070 pounds of leaf meal of 8 percent moisture, equivalent to 1905 pounds on a moisture-free basis, and 1540 gallons of alcohol having a strength of approximately 70 percent. At the completion of an extraction cycle, much of the alcohol can be recovered from the marc by introducing open steam under the false bottom and condensing the vapors driven off.

CONCENTRATION OF EXTRACTS - Each extract (one every 8 hours) is held in a tank of 1500 gallons capacity and is pumped as a continuous feed to the evaporator without filtration. The evaporator would handle each extract as one batch, and the concentrate from that extract would be discharged every 8 hours. The unit, therefore should evaporate dilute alcohol at the rate of approximately 96 gallons per hour. This corresponds to a heating surface of about 20 square feet. This could be either internal or external calandria, either natural or forced circulation, but must be good design. If poor design, or if coils or jacket are used instead of calandria, there should be 30 square feet of heating surface. The evaporator should be of stainless steel and should be provided with condenser and receiver for the recovered alcohol, which may be of copper or stainless steel.

REMOVAL OF TARRY SUBSTANCES - In order to remove most of the fats and tarry materials, the butts from three evaporations are accumulated in two wooden dissolving tanks having a capacity of approximately 800 gallons each. The dissolving tanks are equipped with agitators and means for introducing live steam. Sufficient hot water is added to dissolve the rutin. In order to know how much water must be added to each tank, the approximate quantity of rutin must be known. If the concentrate from each butt is divided equally between the two tanks, the rutin in each will be known, since each tank will contain 95 percent of the total rutin in the buckwheat in one extraction tank. This would be equivalent to approximately 605 gallons of total water in each tank.

The hot rutin solution containing suspended tarry materials is pumped through a steam-jacketed stainless steel filter press having approximately 30 square feet of filter area. To prevent sealing of the pores in the cloths, a precoat of diatomaceous earth should be used. Five pounds per hundred square feet of filter area is satisfactory. A light twill can be used for this filtration, but if it is desired to minimize contamination of the cloths they can be faced with a fast filter paper, which can be discarded after use. The filtration can be conveniently conducted on the basis of two cycles for the liquid in each of the 800-gallon tanks.

PURIFICATION OF RUTIN - From this point on, the operations are with one exception essentially the same as those required for the purification of rutin obtained by the hot-water extraction process. The exception necessitates giving the nearly pure dried rutin three extractions with cold benzol to remove the last traces of benzol-solubles not eliminated in tar filtration. This operation can conveniently be carried out by using for each

extraction 1 gallon of benzol for each pound of dried rutin. It should be done in a closed stainless steel agitated container having a false bottom over which a fine filtering medium, such as cloth, can be fastened. After each extraction, the benzol is withdrawn by vacuum to a receiver. The extracted rutin can be removed from the dissolving chamber through a suitably located hand hole. The benzol can be recovered by distillation. The rutin can be dried in the same drying equipment used for the previous drying step.

Although the procedure for rutin purification is the same in both processes, with the exception of the benzol extraction just mentioned, the crystal filter press which in the hot water process had 100 square feet of filtering area need have only 25 square feet in the dilute-alcohol extraction process, but the frames must be at least 1 inch thick.

YIELD OF RUTIN - The overall yield of pure rutin based on the rutin content of the leaf meal has been conservatively assumed to be 70 percent. Actual pilot-plant and laboratory experiments show that with careful operation overall yields between 75 and 80 percent may be obtained. Factors contributing to good yields include pumping off as much extract as possible from each leach, allowing sufficient time for complete crystallization, and avoiding prolonged heating of alcoholic solution of rutin.

The extraction cycle recommended should remove about 95 percent of the rutin from the meal. The principal losses occur in the mother liquors from the three crystallizations. It is largest in the first mother liquor, where the solubility of rutin in the cold is probably increased by sugars. Attempts to recover additional rutin by evaporation of the mother liquors have not been entirely successful.

HOT-WATER AND ALCOHOL SUPPLY SYSTEMS - The same facilities must be provided for supplying hot water in the dilute-alcohol extraction process as in the hot-water extraction, except that in the former case no supply is needed for extraction proper. The alcohol used in extraction is prepared by mixing weaker alcohol recovered from the marc with stronger alcohol recovered by evaporating the extracts. Facilities should consist of two steel storage tanks equipped with agitators and having a capacity of 1800 gallons each and an aluminum or other rust-resistant tank equipped with an agitator and having a capacity of 1200 gallons. One of the two large tanks should be used for storing alcohol recovered by evaporating the extracts and the other for preparing the 70 percent alcohol for the leaching system. The smaller tank is used to store the dilute alcohol recovered from the marc.

Much of the 95 percent alcohol used in the removal of alcohol insolubles can be recovered by simple evaporation. The portion recovered at a lower strength should be utilized in the extraction process eliminating the need for any rectification equipment. It may occasionally be necessary to concentrate some of the weaker alcohol by simple distillation in one of the existing evaporators in order to balance the alcohol strength.

Suggested List of Equipment

	COST
EXTRACTION TANKS - Two wooden tanks, 6 feet in diameter by 9-1/2 feet deep, with false bottoms covered with 30-mesh stainless steel screen. Two thousand gallons capacity each. Equipped with perforated steam coil under false bottom, and tightly fitting covers. One condenser serves both tanks. Steam is introduced at the conclusion of extraction to strip out alcohol in the marc. 2 at \$625	\$1,250
POSITIVE DELIVERY ROTARY PUMP - Pump for discharging leaching tanks. Must discharge 15 gallons of dilute alcohol per minute.	250
HOLDING TANK FOR EXTRACTS - Wooden tank, 6 feet in diameter by 7 feet deep. Fifteen hundred gallons capacity.	400
EVAPORATOR - Stainless steel evaporator with 20 square feet of evaporating surface. Must evaporate 670 gallons in 7 hours.	2,500
FIRST DISSOLVING TANKS - Two wooden tanks, 5 feet in diameter by 5-1/2 feet deep. Eight hundred gallons capacity each. Agitated. Heated with live steam. Used for hot water to dissolve the rutin in the butts from the evaporator. 2 at \$425	850
FILTER PRESS FOR REMOVING TARS - Stainless steel, steam-jacketed filter press with 30 square feet of filtering surface. No facilities for cake washing required.	1,800
FIRST CRYSTALLIZING TANKS - Three wooden tanks 6 feet in diameter by 6-1/2 feet deep. Fourteen hundred gallons capacity. Equipped with stainless steel coils having sufficient surface to cool the contents from 212° to 80° F. in 2 hours. Agitated for rapid cooling. 3 at \$900	2,700
CRYSTAL FILTER PRESS - Stainless steel backwashing type filter press with 25 square feet of filtering area. Used for 7-1/2 hours each day for filtering first rutin crystals, 6 hours (3 cycles, 2 hours per cycle), for filtering second rutin crystals, and 5 hours for filtering final rutin crystals. This schedule permits an interval of 5-1/2 hours for the thorough cleaning of the press which is necessary before using it for final crystals.	2,000
SECOND DISSOLVING TANK - Tank of same size and construction as first dissolving and crystallizing tanks.	900
SECOND CRYSTALLIZING TANKS - Two tanks of same size and construction as first crystallizing tanks. 2 at \$900	1,800
FILTER PRESS FOR HOT FILTRATIONS - Stainless steel, steam-jacketed filter press with 40 square feet of filtering surface. Used for filtering hot water solutions of the first and second crystals and also later for filtering hot alcoholic solutions of rutin and removing alcohol insolubles.	2,500

	COST
TANK FOR DISSOLVING RUTIN CRYSTALS IN ALCOHOL Stainless steel, jacketed kettle, 3 feet in diameter by 4 feet deep, with reflux condenser. Two hundred gallons capacity. Agitated.	\$1,500
DISSOLVING TANK FOR CRYSTALS IN THE BUTTS AFTER REMOVAL OF STRONG ALCOHOL - Wooden tank, 5-1/2 feet in diameter by 5-1/2 feet deep, with stainless steel coil. One thousand gallons capacity.	800
TANK FOR THIRD (FINAL) CRYSTALLIZATION - Stainless steel, jacketed tank, 5-1/2 feet in diameter by 5-1/2 feet deep. One thousand gallons capacity. Agitated.	3,000
SYSTEM FOR SUPPLYING HOT WATER FOR DISSOLVING CRYSTALS FOR FIRST AND SECOND CRYSTALLIZATIONS Wooden tank, 6 feet in diameter by 5-1/2 feet deep. Eleven hundred gallons capacity. Heated with live steam and equipped with automatic temperature and level control.	400
SYSTEM FOR SUPPLYING CLEAN, FILTERED, HOT WATER FOR DISSOLVING CRYSTALS FOR FINAL CRYSTALLIZATION - Wooden tank, 5-1/2 feet in diameter by 5-1/2 feet deep. One thousand gallons capacity. Heated with stainless steel coils. Equipped with delivery pump having a stone filter in discharge line and with automatic temperature and level controls.	1,200
TRAY DRIER FOR RUTIN BEFORE BENZOL EXTRACTION - Same drier used for drying finished rutin may be employed.	600
BENZOL EXTRACTION UNIT - Stainless steel vacuum filter chamber, with explosion-proof motor. Fifty gallons capacity. Agitated. Should have good ventilation and large hand hole. For three successive extractions using 40 gallons of benzol each.	1,000
STORAGE TANK FOR ALCOHOL RECOVERED FROM EVAPORATION OF LEACHES - Steel tank, 6-1/2 feet in diameter by 7-1/2 feet deep. Eighteen hundred gallons capacity. Agitated.	500
TANK FOR PREPARING 70.0 PERCENT ALCOHOL - Steel tank, 6-1/2 feet in diameter by 7-1/2 feet deep. Eighteen hundred gallons capacity. Agitated.	550
STORAGE TANK FOR WEAK ALCOHOL RECOVERED FROM MARC - Aluminum tank, 6 feet in diameter by 6 feet deep. Twelve hundred gallons capacity. Agitated.	550
SMALL EVAPORATOR - Stainless steel vacuum evaporator having 10 square feet of evaporating surface; condenser and receiver may be copper. Used to replace 95 percent alcohol by water in the alcohol-insoluble removal step. Also used to recover benzol.	1,750
BENZOL STORAGE TANKS - Two steel tanks, 250 gallons capacity each. For storage of used and distilled benzol. 2 at \$100	200

CONCLUSIONS

Pilot-plant operations have demonstrated the feasibility of producing rutin at any season of the year from dried buckwheat leaf meal by either the hot-water extraction process or the dilute-alcohol extraction process. The advantages of each are given below. In the last analysis a choice between the two methods may well depend upon the facilities available in a pharmaceutical plant already established.

In our pilot-plant work, on which the data and suggestions in this circular are based, all extractions of leaf meal were done batchwise, not countercurrent; time was not available to make countercurrent studies. That is, a fresh lot of water (or alcohol, as the case may be) was used for each of the three successive treatments of a batch meal. If countercurrent extraction is found feasible, the evaporation of water in the hot-water process will be reduced to one-third. Similarly, the amount of alcohol in cycle in the dilute-alcohol extraction of leaf meal will be reduced to 45 percent, and the amount in the alcohol extraction of green plant reduced to approximately one-half.

Advantages of the Hot-Water Process

1. No alcohol is employed for the extraction of rutin from the leaf meal thereby eliminating the necessity at this stage for the precautions required with volatile solvents, nor is it necessary to recover alcohol from the marc. In the entire process only 17 gallons of 95 percent alcohol is recycled per pound of rutin produced, including the alcohol used in washing the filter cake. Of this, approximately 2.5 gallons can be recovered without rectification, the rest by rectification. This is significantly less than the 74 gallons per pound of rutin recycled for the dilute-alcohol process, none of which requires rectification, and the 128 gallons per pound of rutin required for green extraction, 73 gallons of which must be rectified.
2. A complete extraction cycle with hot water requires 3 hours, as compared with from 36 to 48 hours when dilute alcohol is used without circulation, or 24 hours with circulation.
3. The necessity for using benzol or other hazardous solvent for removal of fats from the rutin is entirely eliminated, as these materials remain in the marc. Their absence in the purification process makes for improved cleanliness of equipment.
4. No large mixing and storage equipment is required for alcohol.

Advantages of the Dilute-Alcohol Process

1. The number of operators required would probably be less than for hot-water extraction, principally because of a reduction in filter dressing operations. Correspondingly less filter area is required.
2. The evaporation area required for concentrating the extracts is about 20 square feet as compared with 75 square feet required for hot-water extraction.